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Tensile Creep Behavior of Single Fibers and Paper in a Cyclic Humidity Environment

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ABSTRACT

The accelerated creep of paper in a cyclic environment is a well known and complex phenomenon. Even with the extensive amount of work accomplished in this area, the mechanisms responsible for the increase in creep rate have not been established. Student research at the Institute of Paper Science and Technology (IPST) has focused on the role of single-fiber behavior on the accelerated creep of paper, and results of these studies are presented.

Previous single-fiber creep tests conducted at IPST have shown that the tensile creep rate in a cyclic humidity environment did not exhibit any measurable increase in the rate of creep compared with the rate of creep at constant high humidity. The new tests verify this observation, and show that paper made from the same fibers does exhibit accelerated creep. The new tests were completed so that the amount of accelerated creep could be quantified directly from a single test of one fiber; thus, the influence of fiber-to-fiber variability in establishing the presence of accelerated creep was avoided. A single fiber was exposed to a high humidity environment and immediately followed by a cyclic humidity environment. In all tests, no appreciable change in the global rate of deformation was observed. Handsheets were prepared from the fiber source, and creep tests were performed on the single fiber testing apparatus. Results for the paper showed a substantial accelerated creep.

Observations from the current and previous test results are discussed, as well as, their implications on the mechanism for accelerated creep. One possible explanation is that the accelerated creep is a result of nonlinear creep combined with a redistribution of stresses occurring during each cycle of moisture.

Portions of this work were used by S.B.B as partial fulfillment of the requirements for the M.S. degree at the Institute of Paper Science and Technology.

INTRODUCTION

Researchers at IPST have undertaken a renewed effort to understand the basic mechanisms of accelerated creep. In part, this was a result of the work completed by two students at IPST. Kelly Sedlachek (Ph.D., 1995) investigated the creep of single fibers, and Scott Boese (M.S., 1996) investigated the creep behavior of both single fibers and handsheets. Both students used holocellulose fibers from loblolly pine, and conducted tensile tests in both constant and cyclic humidity environments (Slide 1). Sedlachek first observed that the single fibers did not exhibit accelerated creep. In other words, the creep in a cyclic humidity environment was not greater than the creep in a constant high humidity environment. The fiber-to-fiber variability in Sedlachek's results was quite large. Therefore, Boese conducted creep tests on single fibers exposed to an initial period of constant high humidity followed by a period of cyclic humidity. The increase in the slope of the deformation versus log time provided a measure of accelerated creep. Boese performed similar tests on specimens from handsheets prepared from the same fiber source.

Boese's results verified the findings of Sedlachek, and added the observation that the handsheets did exhibit accelerated creep. The present paper presents these findings and provides a discussion as to why the difference in creep behavior between the fiber and handsheets may exist. In addition, a simple mechanism in which accelerated creep is a natural consequence is put forth.

Sedlachek made several noteworthy observations (Slides 2, 3). These are as follows:

- There was no discernible increase in the rate of creep under conditions of cyclic humidity versus constant humidity.
- If a cyclic test started at low humidity, the total creep was greater, but the additional creep occurred in the first cycle.
- Upon moisture sorption, the fibers shortened in the axial direction, and upon moisture desorption, the fibers elongated.

A reasonable explanation of the second observation given above is that the additional observed deformation in the test started at low humidity is due to a release of defects that occur during the first remoisturization of the fibers. For the tests started at high humidity, much of this deformation would have occurred before the creep load was applied and measurements were taken. For the test started at low humidity, the additional deformation would occur during the first increase to high humidity and be included in the creep measurements. A better understanding of the deformation that occurs during the first increase in humidity may provide better insight into box performance in a service environment, which also shows larger deformations associated with the first increase from low to high humidity.

The third observation listed above is most likely a result of the fibular structure of the fibers. The tested fibers had a high fibril angle [≈30°]. For a cylinder of helically wound fibrils that are inextensible, an increase in the cylinder's diameter will cause a decrease in the length. This decrease is proportional to the tangent of the angle. For the pulp fibers, the fibrils are relatively stiff (negligible extension) compared to the amorphous and semicrystalline material surrounding the fibrils. Sorption of moisture causes swelling of this matrix material, and results in radial swelling. The large radial most likely causes the fibrils to rotate and produce the axial shortening observed in the tests. (Slides 4, 5)

The first observation listed above was not easily explained and, due to the large variability in results for single fibers, needed to be verified. In addition, it had to be established whether the fibers tested by Sedlachek produced a paper that exhibited accelerated creep. Thus, the motivation for the present study. (Slide 6)

MATERIALS (Slides 7 and 8)

The tests conducted by Boese were completed with the same fiber source used by Sedlachek. The fibers were obtained from the 18th summerwood growth ring of a 25-year-old plantation-raised loblolly pine (*Pinus taeda L.*). The log was hand-chipped, and the wood chips were converted to fibers by holopulping with sodium chlorite and acetic acid.

The fiber width and lumen diameter were measured for 100 fibers using 400x magnification and image analysis. The fiber diameter was found to be ≈30 mm, and the lumen diameter was ≈13 mm. Thus, the cell wall thickness was estimated to be ≈9 mm.

The average fibril angle of several pulp samples was measured at PAPRICAN using mercury impregnation and viewed through a polarizing microscope with vertical illumination. The fibril angle was found to be ≈30°.

A handsheet was prepared from this same fiber source, by beating the hollocellulose fibers in a PFI mill for 500 revolutions (CSF = 640). A 1.2-gram sheet was formed in the British handsheet mold, pressed and air dried under restraint.

CREEP TESTS (Slides 9, 10)

The creep tests for both the single fibers and handsheet specimens were conducted in a load-controlled tensile tester constructed at IPST (Slide 8). Using epoxy, the samples were mounted in the tester. The specimen is kept in a controlled humidity chamber, which was programmed for a specific humidity history.

For the present study, the handsheet specimens had a width of 1 mm and a length of 13 mm. The fiber span was about 1 mm. The creep load applied to the fibers was 10 gm, and the creep load applied to the handsheet specimens was 30 grams. It was found that the displacement history for samples with loads lower than 10 grams had large fluctuations. The maximum load for the load cell was 50 grams.

The humidity history for the creep tests consisted of an initial period of creep for 2 hours at a constant high humidity of 90% RH followed by a cyclic humidity period where the humidity was varied between 90 and 20% RH. A complete cycle of humidity was 20 minutes.

DISCUSSION OF RESULTS

Boese results confirmed that the single fibers did not show accelerated creep, and that the fiber shortened upon moisture sorption. For the handsheet specimens, Boese found substantial accelerated creep. As expected, the paper expanded upon sorption of moisture. The degree of accelerated creep was judged by the difference in slope of the elongation versus log of time curve from the constant humidity regime and the cyclic humidity regime. Thus, a degree of accelerated creep can be determined for each specimen, and the large variability in creep rate for single fibers does not come into play. (Slides 11,12)

The initial response to the test results is that bond breaking must be responsible for accelerated creep, but this conclusion may be premature. (Slide 13). A closer look at the results is warranted. For the fibers tested, the slope of the average creep rate in cyclic humidity to the average ratio of the creep in constant humidity was found to be 1.1. This is not significantly different than one, and thus, the conclusion is that the fibers do not show accelerated creep. In fact, for some fibers, the ratio of slopes was actually less than one. The degree of accelerated creep for the handsheet specimens is observed by the high ratio of slopes; 7.2 for the ratio of the averages. (Slides 14-17)

The first notable difference between the single fibers and handsheets is the difference between the physical state of single fibers and fibers in the sheet. The single fibers were dried flat and straight, and would have few defects or microcompressions dried into the fiber. The fibers in the sheet will be influenced by the radial shrinkage of neighboring fibers, and will form microcompressions at or near the bonds. These microcompressions or defects may lead to the observed accelerated creep in paper. (Slide 18)

The second difference between the single fibers and handsheets is the time needed for moisture sorption. Sedlachek found that moisture sorption occurred on the order of seconds. In fact, the actual time was smaller than the time needed for the instrument to become stable, and therefore, the time could be quite small. Sorption of moisture into the paper samples occurred on the order of minutes. Thus, the extra time while the moisture is changing in the paper specimens could contribute to the observed accelerated creep. (Slide 19)

A third difference in the tests of single fibers and handsheets was the rate of creep. Although the deformation rates were similar, the strain rate was much smaller for the paper samples as compared to the fiber. This is because fiber length (1 mm) was much shorter than the length of the paper sample (13 mm). Under constant 90% RH, the average strain rate of the single fiber was 20 times larger than the average strain rate of the handsheets. Under cyclic humidity conditions, the strain rate was still three times larger for the fibers than the handsheets. Single fibers tested under a load of 5 grams did not produce useful results because the fluctuation in load was too large during the tests. It would be of interest to investigate the effect of creep strain rate on the degree of accelerated creep. (Slide 20)

The big unanswered question is still what leads to accelerated creep. At IPST, a project is currently underway to better understand the mechanisms of accelerated creep. D. Coffin and C. Habeger have developed a simple mechanism that is not at odds with the present experimental results. In short, it is proposed that accelerated creep is due to nonlinear creep behavior coupled with a redistribution of stresses that occurs with each cycle of moisture. This redistribution of stresses arises because of either constantly changing moisture gradients or material heterogeneity. For this mechanism to work, both the nonlinearity in creep rate versus load and a continuous change in stress are essential. Because the material response is nonlinear, the creep response to a distributed load will be different than the response to the average load. Thus, it is quite easy to get higher levels of creep in a state of continuous stress redistribution than when the stress is uniformly distributed in the most compliant state. With this mechanism, accelerated creep is easily explained and does not require any extraordinary phenomena to explain it. Thus, creep under constant humidity is no different than creep at constant humidity, except that the changes in the stress distribution produce different amounts of creep. (Slides 21-23)

In light of this mechanism, several interpretations for the differences between the single fiber and handsheet response can be made. It is possible that the fiber sorbs moisture so quickly that there is no time for accelerated creep to occur. The accelerated creep of the single fiber may be masked by the high rates of creep in the test: the changes in stress are too small to produce a detectable change in creep. It is possible that because of the structural differences in the single fiber and the fibers in the sheet the nonlinearity of the creep is greater in the paper than in a straight single fiber. Finally, the moisture gradients or material heterogeneity in the paper may produce larger stress gradients as compared to those in the fiber. (Slide 24)

Thus, it is concluded that other plausible explanations rather than bond breaking may cause accelerated creep. The preceding results and discussion give rise to several research needs. First, taking the view of accelerated creep given above, it is of interest to assess the degree of accelerated creep as a function of load. Would a single fiber under lower load show accelerated creep? Would a paper specimen under higher load not show as much accelerated creep? Second, the role of rate of moisture sorption needs to be clarified. The mechanism suggests that the amount of creep would increase if the time in which stress gradients is extended. Third, it would be of interest to assess the effect of microcompressions, dislocations, and defects on accelerated creep of paper. (Slide 25)

Title Slide

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Slide 1

Student Work at IPST

- **Kelly Sedlachek (Ph.D., 1995)**
  - **Investigated the tensile creep of loblolly pine holocellulose fibers subjected to constant and cyclic humidity**
- **Scott Boese (MS , 1996)**
  - **Investigated the accelerated creep of loblolly pine holocellulose fibers and handsheets in a cyclic humidity environment**

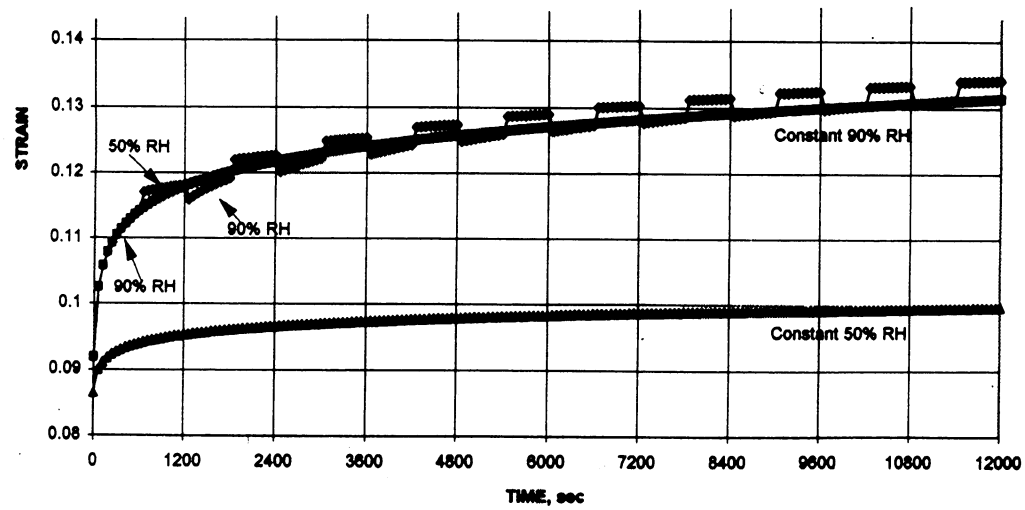
Slide 2

Results found by Sedlachek for Single Fiber Creep

- **There was no discernible acceleration in the rate of creep under conditions of cyclic humidity versus constant high humidity.**
- **If cyclic test started at low humidity, total creep was greater, but additional creep occurred in first cycle.**
- **The fibers shortened in the axial direction upon the sorption of water.**

Slide 3

Creep of Single Fibers: Constant Rh Compared to Cyclic Rh



(Sedlachek, 1995)

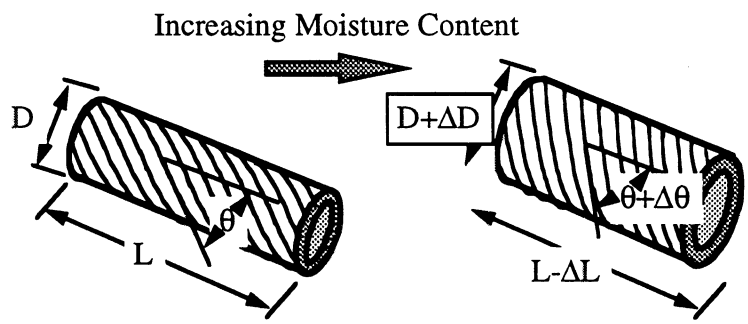
Slide 4

Hygroexpansion of Single Fibers

- **The tests showed that during the sorption of moisture the fibers swell in the radial direction, and shrink along the fiber axis.**
- **This is most likely due to the high fibril angle of the fibers.**
- **Radial swelling would tend to increase the fibril angle, and consequently shorten the fiber along the axis.**

Slide 5

Hygroexpansion of Single Fibers



Helix with constant arc length

$$\frac{dL}{dD} \propto -\mathrm{Tan}(\theta)$$

Slide 6

Objective of Research

- **Verify the results of Sedlachek.**
- **Determine if handsheets made from same fiber source exhibited accelerated creep.**

Slide 7

Fiber Source

- **18th summerwood growth ring of a 25 year-old plantation-raised loblolly pine.**
- **Holopulped using sodium chlorite and acetic acid.**
- **Fibril angle ≈30°, fiber diameter ≈30μm, fiber width ≈9μm.**

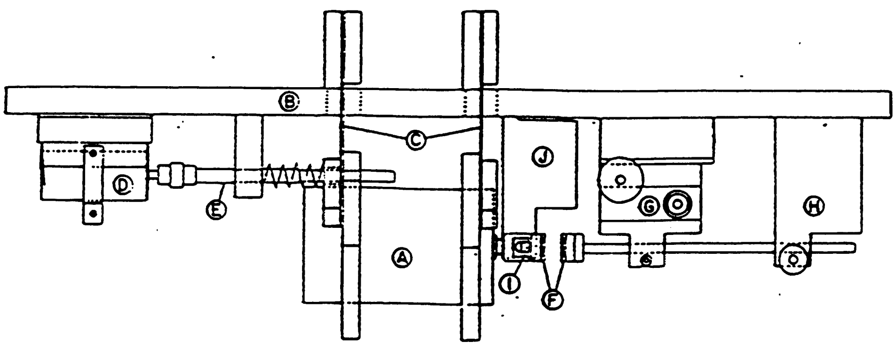
Slide 8

Sample Preparation

- Individual wet-fibers isolated and air-dried under restraint.
- Handsheets prepared by beating fibers in a PFI mill (500 revolutions). A 1.2 gram sheet formed in British Handsheet mold, pressed, and air dried under restraint.

Slide 9

Tensile Testing Apparatus



Scale drawing of apparatus. A: electronic weighing cell. B: mounting plate. C: flexure hinges D: DC servo motor. E: differential screw F: pin holder clamps. G: microscope focusing mechanism. H: pillar clamp. I: capacitive displacement transducer. J: support pillar for displacement transducer.

Slide 10

Test Conditions

- Sample ends restrained with epoxy.
- Constant creep load applied to test specimen.
  - 10 grams for fiber
  - 30 grams for handsheet
- Humidity of environment controlled with prescribed Rh.
  - 90 to 20% Rh cycle

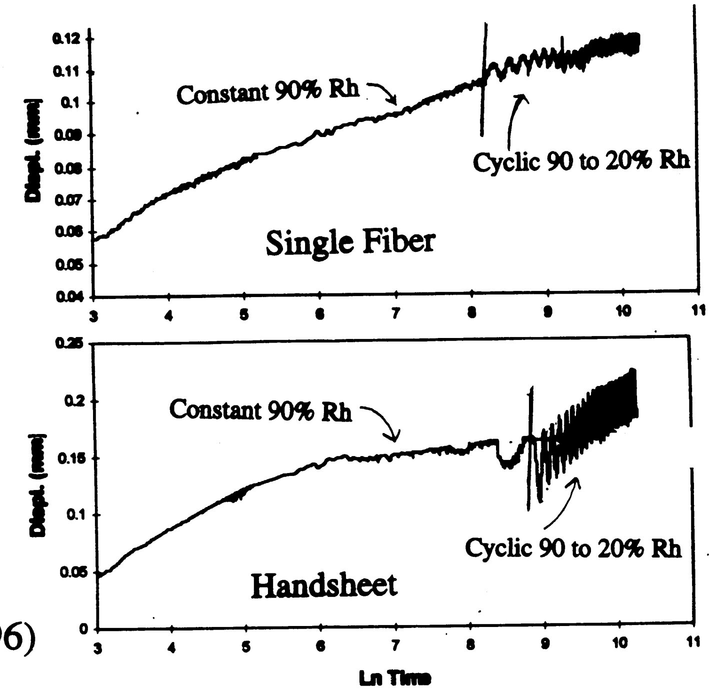
Slide 11

Results for Creep of Single Fibers and Handsheets

- Did not observe accelerated creep in single fibers.
- Observed substantial accelerated creep in handsheets.
- Fiber length shortens upon sorption of moisture.
- Paper expands upon sorption of moisture.

Slide 12

Creep of Single Fibers Compared to Creep of Handsheets



(Boese, 1996)

Slide 13

Is Bond Breaking Responsible for Accelerated Creep ?

- **Immediate reaction: breaking of fiber-to-fiber bonds leads to accelerated creep!**
- **This is a premature conclusion and likely not the cause of accelerated creep.**

Slide 14

Comparison of Creep Rates in Fibers and Handsheets

Creep Deformation Rates [mm/ln(time [sec])]

Specimen	Constant 90%	Cyclic 90%/20%	Ratio
Single Fiber	0.0045	0.0050	1.1
Handsheet	0.0029	0.0209	7.2

Creep Strain Rates [%/ln(time [sec])]

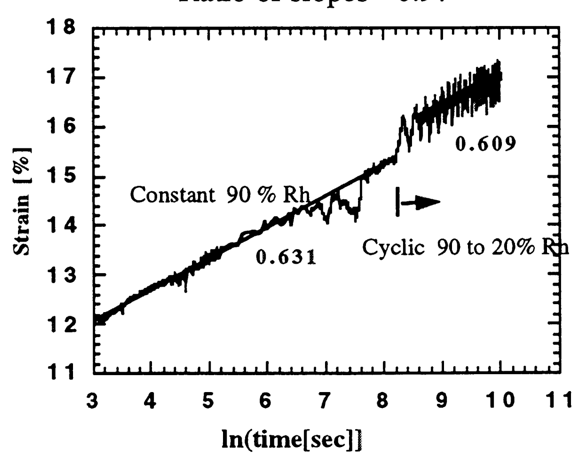
Specimen	Constant 90%	Cyclic 90%/20%	Ratio
Single Fiber	0.45	0.50	1.1
Handsheet	0.022	0.161	7.2

fiber span was 1mm
handsheet specimen span of 13mm, width of 1 mm

Slide 15

Typical Response of Fiber

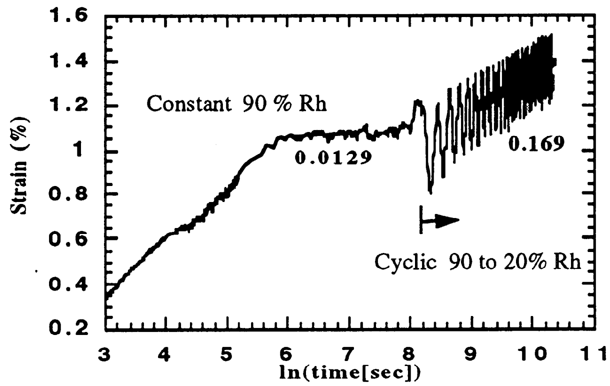
Ratio of slopes =0.97



Slide 16

Typical Response of Handsheet

Ratio of slopes =13.1



Slide 17

Change in Deflection between High and Low Humidity

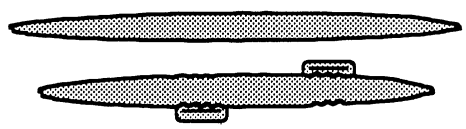
Change in deformation going from 90% Rh to 20% Rh

Specimen	Δu [mm]	$\Delta\varepsilon$(%)
Single Fiber	**0.0045**	**0.45**
Handsheet	**-0.0324**	**-0.25**

Slide 18

Differences Between Single Fiber and Handsheet Results

- **Single fibers were dried straight, no "microcompressions"**
- **Fibers in paper will form "microcompressions" at bond site**



Slide 19

Differences Between Single Fiber and Handsheet Results (cont.)

- **Sorption of moisture in single fibers found to be of the order of seconds by Sedlachek, (1995)**
- **Sorption of moisture into handsheet occurs on the order of minutes.**

Slide 20

Differences Between Single Fiber and Handsheet Results (cont.)

- **Strain rate of creep in single fibers tests higher than in handsheet tests.**

Ratio of single fiber to handsheet creep rate

Constant 90%	Cyclic 90%/20%
20	3

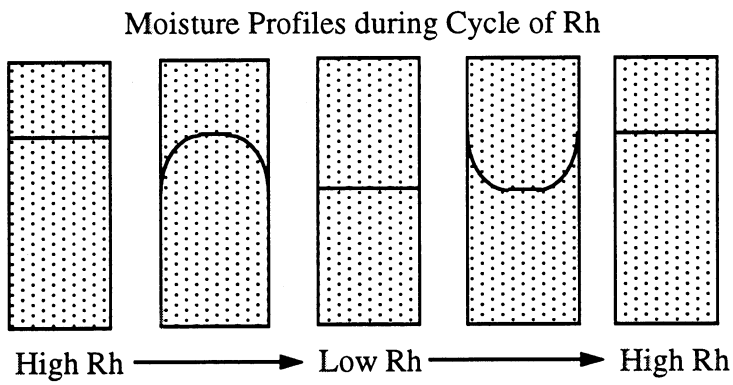
Slide 21

Explanation for Accelerated Creep

- **Accelerated creep is due to the nonlinear creep behavior coupled with a redistribution of stresses during each cycle of humidity**
  - **Redistribution of stresses occurs because of**
    - **constantly changing moisture gradients**
    - **material heterogeneity**

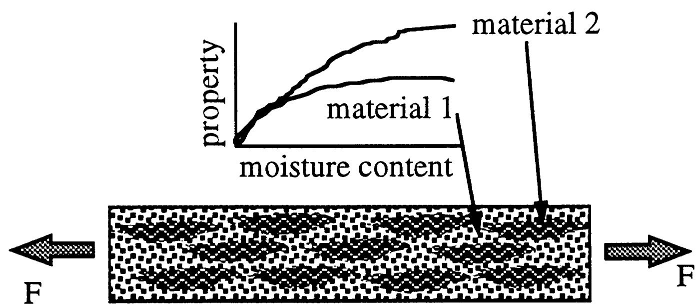
Slide 22

Cyclic Moisture Gradients



Slide 23

Material Heterogeneity



If the distribution of material properties change during the moisture cycle, the stress distribution in the sheet will change.

Slide 24

Interpretation of Results for Single Fiber Creep

- Fiber sorbs moisture quickly; thus, short time for accelerated creep.
- High creep rate of single fiber tests masks accelerated creep.
- Nonlinearity of creep is greater in paper than for a straight single fiber.
- Magnitude of change in local stress greater in paper than fiber.

Slide 25

Research Needs Arising from Present Study

- Assess the degree of accelerated creep as a function of creep load.
- Assess the role of moisture sorption rate on accelerated creep.
- Assess the role of "microcompressions", dislocations, and defects on the role of accelerated creep of paper.